

**Comparison of the storage stability of starch and pectin black  
raspberry confections**

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Undergraduate Research Thesis

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## **Abstract**

Black raspberries (BRB) are a rich source of bioactive phytochemicals and nutrients, such as anthocyanins and ellagitannins. The phenolic compounds in BRB have drawn much attention recently due to their promising anticancer effect. Physicochemical properties such as texture and water content/activity may influence the phenolic compound stability and sensory acceptance during storage. Therefore, finding suitable food matrix with extended shelf life, higher physicochemical stability and production consistency is important in delivering the bioactive compound from BRB in clinical trials. The objective of this study is to assess the physicochemical stability of two different BRB matrices: Starch and pectin based gummies, under different storage temperature conditions for two months. Total water content, water activity ( $A_w$ ), texture, and rheological properties were measured for each type of confection during two-month storage under room temperature (RT) and 4 °C. All samples were selected randomly from three batches of scale-up productions with replicates for each test. There was no significant change in elasticity ( $G'$ ) for pectin stored at both RT and 4 °C. However, for starch gummy, room temperature storage had a significant increase in elasticity compared with fresh sample ( $P < 0.05$ ). Lower temperature storage overall reduced the water loss in starch confection, showing no significant change even after two months ( $P > 0.05$ ). However, the total water content of pectin gummy changed significantly during the entire storage time for both conditions ( $P < 0.05$ ). Starch confection maintained an  $A_w$  lower than 0.7 during storage, but the pectin gummy had  $A_w$  higher than 0.7, increasing the potential of microbial growth. The results from this study can be used to select storage conditions and suitable food matrix for BRB confections to be used in future human clinical trials.

## **Introduction**

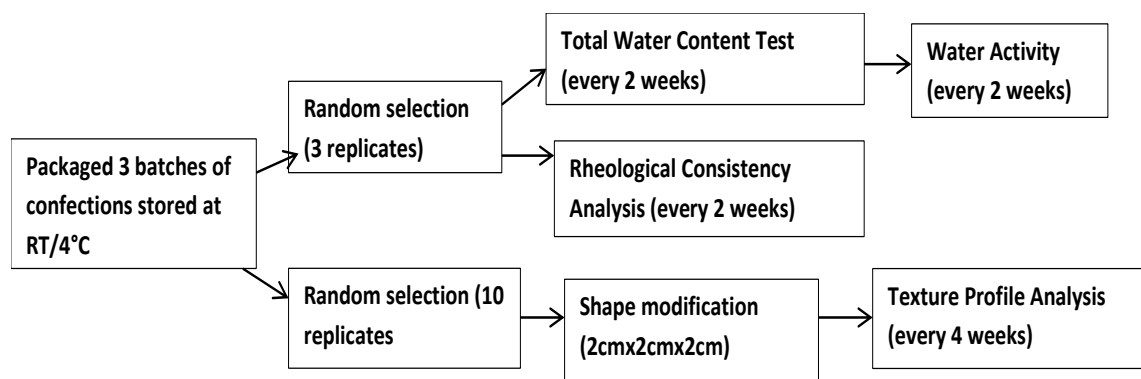
Black raspberries (BRB) have drawn food scientists' attention due to their rich source of bioactive phytochemicals and nutrients, such as anthocyanins and ellagitannins that are antioxidants (Stoner 2009) and have promising anticancer effect, (Cooke and others 2005). As reported, they may be involved in inhibiting chronic inflammatory processes which are associated with the initiation and promotion of cancer in various organs, especially demonstrating anti-inflammatory, antioxidant, and anticancer properties in oral cancer cells (Gu and others 2014). Even though BRB showed promising effect in oral disease prevention, many of the bioactive components consumed from fruit and vegetable are concerned to be poorly absorbed (Epriliati and Ginjom 2012). Previous research demonstrated that a nonfood-based bioadhesive gel containing 5% to 10% freeze-dried whole BRB is an effective delivery system of anthocyanins to saliva and oral tissues (Ugalde and others 2009) for patients with premalignant oral lesions (Mallery and others, 2007, 2008). Additionally, because anthocyanins are known to be unstable compounds during food processing and storage due to their susceptibility to numerous chemical and enzymatic reactions that lead to loss of chemical form and bioactivity (Kadivec and others 2013), a suitable food matrix need to be carefully designed so that these bioactives can be stored, delivered and released at desirable rate. Previous research showed that polymer gels formed by starch or pectin have 3-dimensional network that can protect and entrap these bioactives for targeted delivery (Yu 2001). Total water content, water activity ( $A_w$ ), texture, and rheological properties are some of the most important factors in qualifying physicochemical stability of a product throughout storage. The consistency of these properties may change during product storage and this can lead to serious safety problem, affect quality acceptability and influence the function of delivery. The objective of this study is to

assess the physicochemical stability of two different BRB matrices: starch and pectin based gummies, under different storage temperature conditions for two months.

## **Material and Method**

Pectin-based and starch-based confections were prepared by mixing water, sugar (sucrose, US food service, Cincinnati, Ohio, USA), corn syrup (Gordon Food Service, Springfield, Ohio USA), and gelling agents (Grindsted pectin, 70% esterification, CF 130B, Danisco USA Inc., New Century Kans., USA; Confection G food starch, Tate & Lyle, Decatur, Ill., USA) and stirring the mixture on a stirrer plat to a final temperature of 95°C and °Brix of  $67 \pm 2$ , determined by a hand-held refractometer (range 58-90 °Brix, Fisher Scientific Japn Ltd., Tokyo, Japan). After cooling to 70°C at room temperature, freeze-dried BRB powder (Stokes Raspberry Farm, Wilmington, Ohio, USA) was added and mixed into the gel. The mixture was deposited with a pastry bag and the product were equilibrated at room temperature in the dark for 25 hours before placing each into 2 oz. plastic portion cups with lids.

Packaged 3 different batches of starch and pectin confections were stored at both room temperature and 4°C before any testing. 3 replicates of each kind of confection were random selected and tested for total water content, rheological consistency and water activity every week during a 2 month period. In order to obtain consistent and accurate data, 10 replicates of each kind of confection were random selected and cut into 2cm x 2cm x 2cm cube for texture profile analysis. Texture profile analysis was conducted every 4 weeks during a 2 month period. A flow chart of this testing process is shown in Figure 1.



**Figure 1.** Pectin and starch BRB confections testing flow chart

#### *Total Water Content and Water Activity*

A Thermogravimetric Analyzer, TGA Q-5000, was used to determine the total moisture content of each confections. 15-20 mg sample were selected from the middle section of each replicates and then analyzed with a heating rate of 10.00 °C/min and a final temperature of 200 °C. The rest of samples were used for water activity test.

#### *Rheological Consistency*

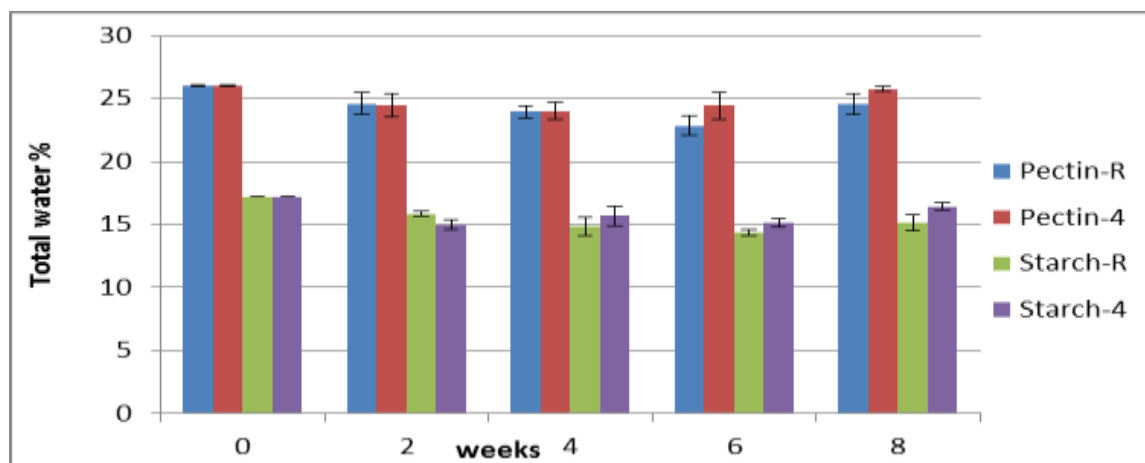
Confections were analyzed with an AR 2000ex Controlled Stress Rheometer with a 20 mm diameter probe. Elasticity ( $G'$ ) was recorded and compared by dynamic frequency sweep (0.1–100 Hz) tests carried out at 25°C with 0.1% strain to obtain viscoelastic behavior of confections.

#### *Texture Profile Analysis (TPA)*

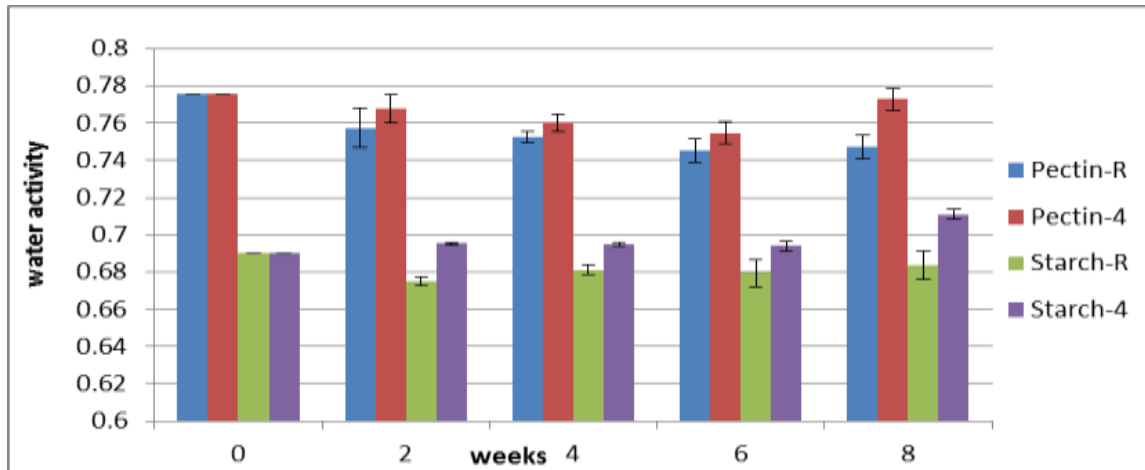
Confections' texture profile analysis was conducted by Instron 5542 Universal Testing Machine, using 35mm compression parallel plate geometry. In order to simulate mastication, TPA with 40% compression at a rate of 1mm/s was used (Daubert and Foegeding 2003). Hardness, springiness, cohesiveness, gumminess and chewiness were measured and analyzed for both confections.

## Results and Discussion

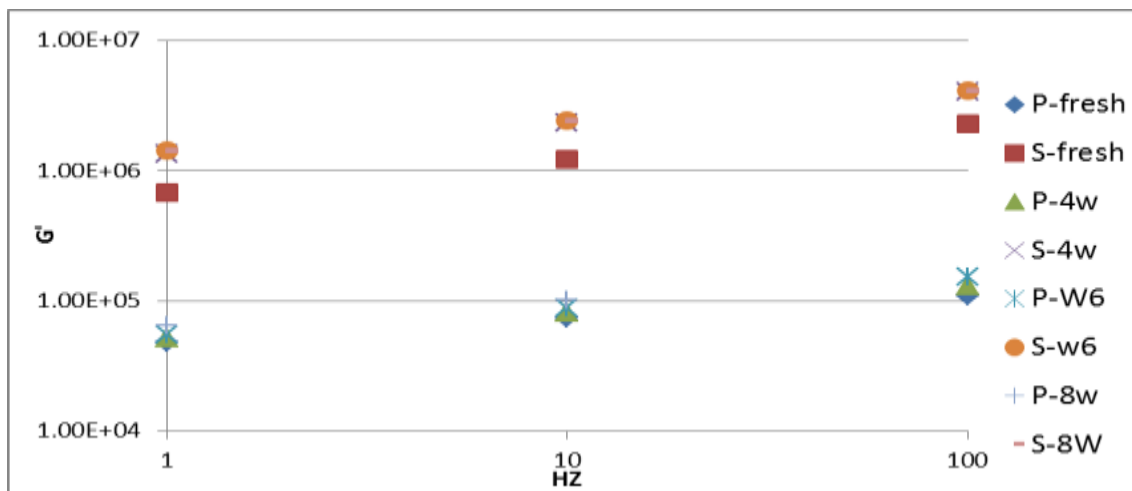
Water activity ( $A_w$ ) is the measure of available water not bound to food molecules and can be used for microbial growth. Lower  $A_w$  is one of the factors that contribute to a longer shelf life. It is well known that foods with  $A_w > 0.85$  are more exposed to spoilage of molds, yeast or bacteria. As shown in Figure 3, starch confection stored at room temperature maintained an  $A_w$  lower than 0.7 during storage, but starch confections stored at 4°C had a significant increase from 0.68 to 0.71. Pectin gummy had comparable higher water activity around 0.77. Pectin stored at room temperature had a significant decrease in water activity during storage but maintained the original water activity at 4°C. As shown in Figure 2, pectin storage at room temperature had significant decreased in total water content from fresh to 4 weeks and 6 weeks ( $p < 0.05$ ). But then the water content increased in 8 weeks, and made no differences than original ( $p > 0.05$ ). Pectin storage at 4°C had significant decreased in total water content from fresh to 4 weeks ( $p < 0.05$ ), but then the water content increased in 6-8 weeks ( $p > 0.05$ ), and made no difference from original sample. Starch gummy storage at 4°C had no significant change after 8 weeks. Starch gummy storage at RT significantly decreased after 4 weeks ( $p < 0.05$ ).



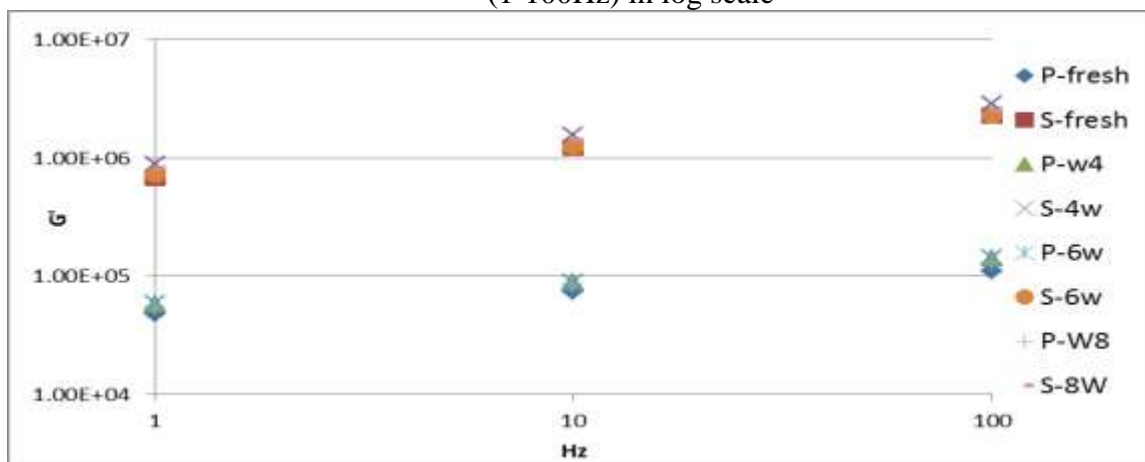
**Figure 2.** Total water content change of Pectin and Starch confections from fresh to 8 weeks



**Figure 3.** Aw change of pectin and starch confections from fresh to 8 weeks

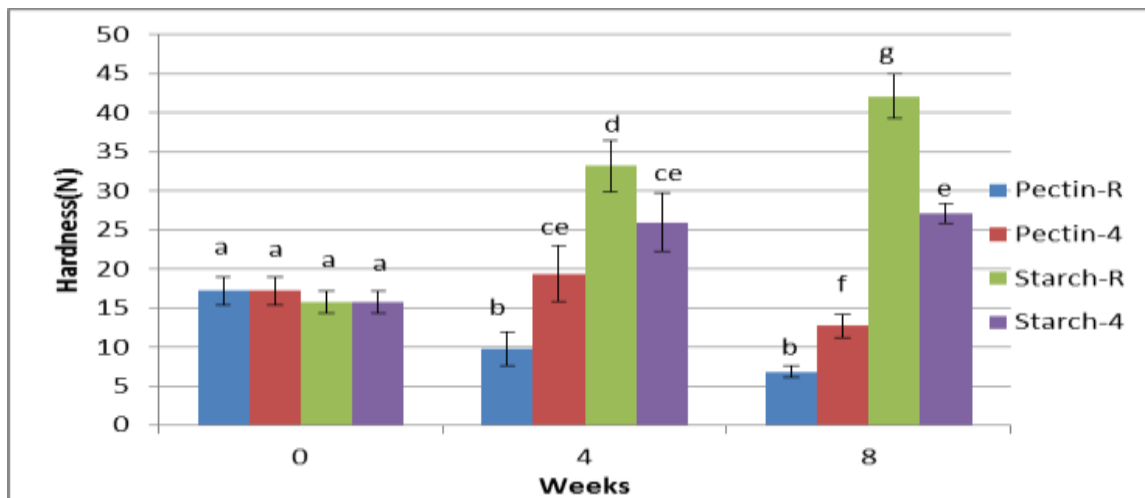


**Figure 4.** Elasticity ( $G'$ ) of pectin vs starch gummy at RT from fresh to 8week- storage tested (1-100Hz) in log scale



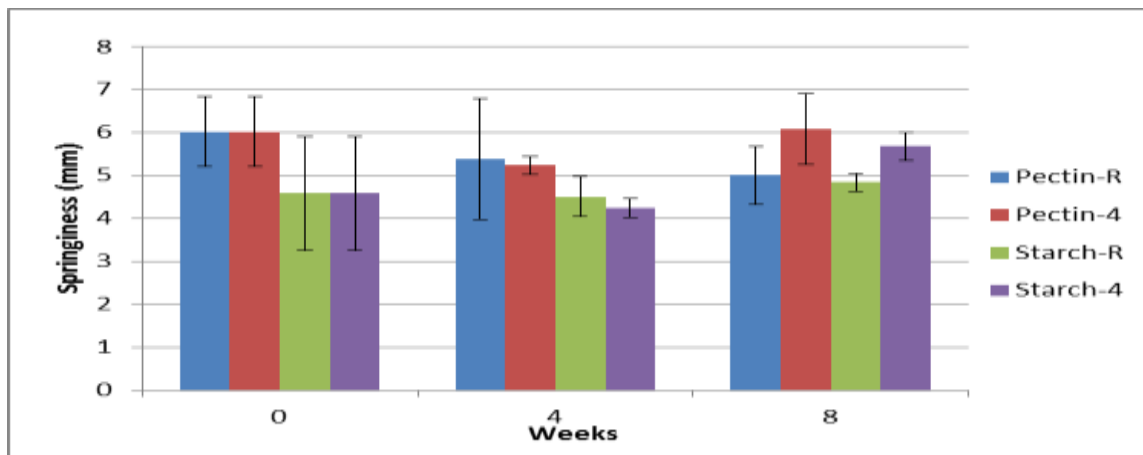
**Figure 5.** Elasticity ( $G'$ ) of pectin vs. starch gummy at 4°C from fresh to 8 week-storage (1-100 Hz) in log scale.

As shown in Figure 4 and Figure 5, increased frequency resulted in an increased  $G'$  for all samples, indicating the confections were weak gels (Tabilo-Munizaga, and Barbosa-Caovas 2004). At both storage conditions, starch-based gummy had significant larger elasticity  $G'$  value than that of pectin-based gummy. At  $4^{\circ}\text{C}$ , there was no significant change in  $G'$  for both starch and pectin gummy during the two- month storage. In contrast, at room temperature storage,  $G'$  of starch confection increased significantly during the first 4 week storage, but no further significant change throughout the rest of storage. Overall, both products showed solid like properties and were comparatively stable throughout the storage.

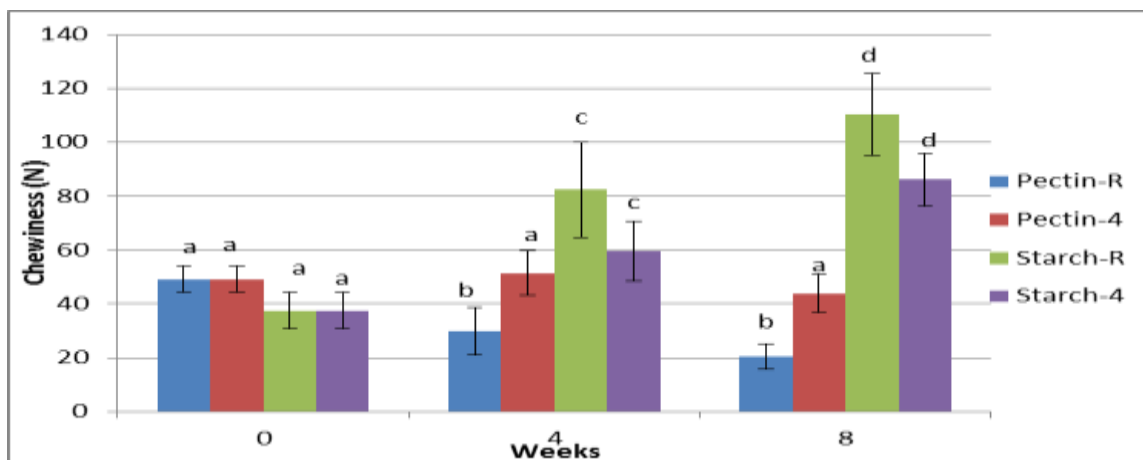


**Figure 6.** The hardness of pectin and starch confections from fresh to eight weeks under room temperature and  $4^{\circ}\text{C}$





**Figure 7.** The springiness of pectin and starch confections from fresh to eight weeks under room temperature and 4°C



**Figure 8.** The chewiness of pectin and starch confections from fresh to eight weeks under room temperature and 4°C

Texture Profile Analysis showed the changes in hardness, springness, and chewiness of both products stored at 4°C and room temperature for two month. From Figure 6 and Figure 8, we can see that there was a significant increase in hardness and chewiness for starch confections stored at RT and 4° by the end of 8 weeks storage compared with fresh samples. There was significant decrease in hardness for pectin gummy stored at both conditions for 2 months. The

chewiness of pectin did not change significantly when stored at 4°C. There was no significant change in springness for both products when stored at both conditions.

## **Conclusion**

There was no significant change in elasticity ( $G'$ ) for pectin stored at both RT and 4 °C. However, for starch gummy, room temperature storage had a significant increase in elasticity compared with fresh sample ( $P < 0.05$ ). Lower temperature storage overall reduced the water loss in starch confection, showing no significant change even after two months ( $P > 0.05$ ). However, the total water content of pectin gummy changed significantly during the entire storage time for both conditions ( $P < 0.05$ ). Starch confection maintained an  $A_w$  lower than 0.7 during storage, but the pectin gummy had  $A_w$  higher than 0.7, increasing the potential of microbial growth. The results from this study can be used to select storage conditions and suitable food matrix for BRB confections to be used in future human clinical trials.

## **Future Research**

Further research should focus on sensory test on each type of product to better understand consumer preferences. Retention of total phenolics and anthocyanins in BRB confections should also be compared for both matrices in order create a stable product with high bioactive compound retention rate.

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